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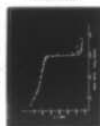
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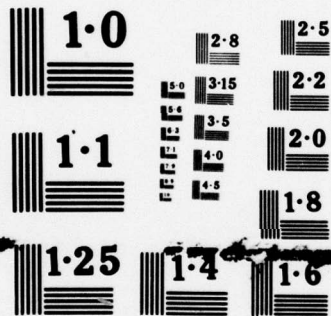
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*9* TECHNICAL REPORT, NO. 8

*14* TR-8

*6* POTENTIOMETRIC INVESTIGATION OF DIALUMINUM HEPTACHLORIDE  
FORMATION IN ALUMINUM CHLORIDE: 1-BUTYLPYRIDINIUM CHLORIDE MIXTURES

by

*10* R. J. Gale ~~and~~ R. A. Osteryoung

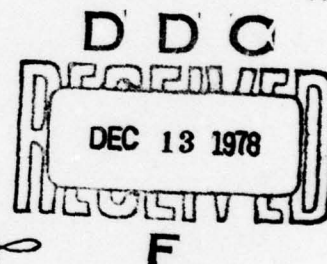
Prepared for publication in

Inorganic Chemistry

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*11* Nov ~~1977~~ 1978



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1. REPORT NUMBER Technical Report No. 8✓	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) "Potentiometric Investigation of Dialuminum Heptachloride Formation in Aluminum Chloride: 1-Butylpyridinium Chloride Mixtures"		5. TYPE OF REPORT & PERIOD COVERED Technical
7. AUTHOR(s) R. J. Gale and R. A. Osteryoung		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Department of Chemistry✓ Colorado State University Fort Collins, Colorado 80523		8. CONTRACT OR GRANT NUMBER(s) N00014-77-C-0004✓
11. CONTROLLING OFFICE NAME AND ADDRESS Chemistry Program Office of Naval Research Arlington, VA 22217		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) Office of Naval Research 1030 East Green Street Pasadena, CA 91106		12. REPORT DATE November 1978
		13. NUMBER OF PAGES 14
		15. SECURITY CLASS. (of this report) Unclassified
16. DISTRIBUTION STATEMENT (of this Report) Approved for Public Release; Distribution Unlimited		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES Prepared for publication in <u>Inorganic Chemistry</u>		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Chloroaluminate melts; Potentiometry; Dissociation constants		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The solvent acid-base properties of $AlCl_3$ : 1-butylpyridinium chloride melts from 2.26/1.0 to 0.60/1.0 molar ratios, respectively, have been investigated by potentiometry. An equilibrium constant $K_f$ for the dissociation reaction.		

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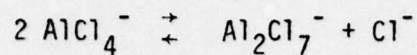
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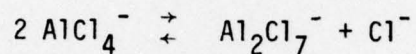
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# ABSTRACT

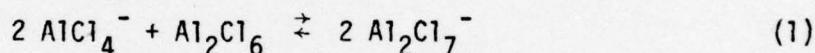
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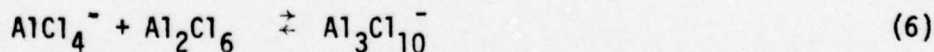
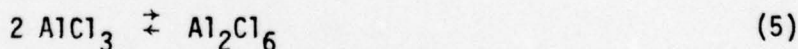
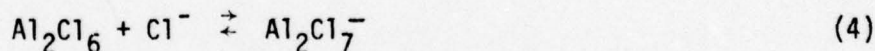
## INTRODUCTION

Considerable interest has been shown recently in molten salt mixtures that are liquid at ambient temperatures, in view of the possibilities for their use as novel matrix solvents or electrolytes. The fused aluminum halide: alkylpyridinium halide systems are low melting, relatively easy to synthesize, and miscible with organic solvents such as benzene<sup>[1-4]</sup>. An investigation of the ionic species equilibria in  $\text{AlCl}_3$ : 1-butylpyridinium chloride mixtures, by Raman spectroscopy<sup>[5]</sup>, has indicated that the association reaction



is virtually complete and that no molecular aluminum chloride could be detected in the 2:1 molar ratio mixture at room temperature.

The following equilibria have been commonly used to relate the major solvent species in  $\text{AlCl}_3$ :MCl systems, M usually being an alkali metal cation<sup>[6-11]</sup>



If an aluminum reference electrode behaves as a reversible indicator electrode, model equilibria may be applied to potentiometric titration data using the Nernst equation,

$$E = E_o + \frac{RT}{3F} \ln \frac{a_{\text{Al}^{3+}}}{a_{\text{Al}(0)}} \quad (7)$$

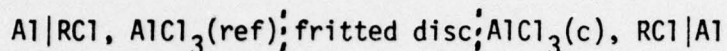
which, incorporated with the association constants for reactions (2) and (3), gives for a reference state (superscript o) and any other state i



(superscript i),

$$\Delta E = \frac{RT}{3F} \ln(a_{\text{AlCl}_4^-}^0 / a_{\text{AlCl}_4^-}^i) + \frac{4RT}{3F} \ln(a_{\text{Cl}^-}^i / a_{\text{Cl}^-}^0) \quad (8)$$

Hence, an electrochemical cell of the type,



where R represents an alkylpyridinium cation, may be used as a p Cl electrode. The purpose of the present investigation is twofold, (i) to quantitatively assess the association constants for the major ionic equilibria, and (ii) to examine the effects that the nature of the cationic moiety of the halide salt have on the solvent equilibria.

#### EXPERIMENTAL

Melts were prepared by mixing accurately weighed quantities of purified aluminum chloride and 1-butylpyridinium chloride. The purification procedures for these compounds are described elsewhere<sup>[5]</sup>. In order to avoid discoloration and thermal decomposition, the pieces of aluminum chloride were added slowly, with stirring, to the 1-butylpyridinium chloride crystals in the cell. The colorless solvents can be titrated either by weighed additions of 1-butylpyridinium chloride to an acidic melt or by anodization of an aluminum electrode to a basic melt, which is tantamount to the addition of  $\text{AlCl}_3$ . The former procedure was finally adopted because of a spontaneous reaction of aluminum with the solvent in melts which contain an excess of free chloride ions, *vide infra*.

The electrochemical cell employed for the potentiometric titrations was made of Pyrex and the reference electrode compartment, containing coiled aluminum wire (m 5N Alfa Products), was isolated with a fritted



Pyrex disc. The electrolyte level in the reference electrode compartment was maintained very slightly higher than that of the bulk solution. A proportional thermoelectric controller was used to control the temperature of the melt to  $\pm 1^\circ\text{C}$  using a Pyrex-sheathed Chromel-Alumel thermocouple (Thermo Electric 400). All operations were made under a drybox atmosphere of purified argon.

### RESULTS

Figure 1 illustrates the results of experimental titrations from approximately 2.0:1.0 to 0.6:1.0 molar ratio  $\text{AlCl}_3$ : 1-butylpyridinium chloride at  $30^\circ\text{C}$ ,  $60^\circ\text{C}$  and  $120^\circ\text{C}$ , respectively. The overall features of the sigmoidal curve are distinct in several respects from those obtained for the  $\text{AlCl}_3$ :  $\text{NaCl}$  melt at  $175^\circ\text{C}$  (6). Firstly, the potential difference between the plateau regions in figure 1 is larger by a factor of approximately 2-3 than that of the  $\text{AlCl}_3$ : $\text{NaCl}$  melt, resulting in a greater p Cl range. Secondly, the data for the basic portion of the curve were less reproducible than those for the acidic regions and a blue coloration appearing at the aluminum electrode is an indication that secondary reaction occurs. Finally, unlike the potential data for the high temperature  $\text{AlCl}_3$ : $\text{NaCl}$  systems, the curve changes slope again in the region of the 2:1 molar ratio melt composition.

#### Estimation of equilibria constants

An approximate conditional equilibrium constant for the potentiometric data of figure 1, in terms of the major equilibrium reaction (1), may be obtained using equation (8). The activities of any free  $\text{Al}^{3+}$  ions,  $\text{Al}_3\text{Cl}_{10}^-$  ions, molecular  $\text{Al}_2\text{Cl}_6$  or  $\text{AlCl}_3$  are disregarded for the purposes of this

calculation with respect to the major ionic constituents of the melt, namely  $\text{AlCl}_4^-$ ,  $\text{Al}_2\text{Cl}_7^-$  and  $\text{Cl}^-$ . The liquid junction potential at the Pyrex frit reference was neglected<sup>[6,7]</sup>. Then, at 30°C,

$$K'_3 = \frac{[\text{Al}_2\text{Cl}_7^-][\text{Cl}^-]}{[\text{AlCl}_4^-]^2} = 3.83 \times 10^{-13}$$

The potential function represented by equation (8) is shown in figure 1 as a solid line for this value of  $K'_3$ . Compared to the  $\text{AlCl}_3\text{:NaCl}$  system at 175°C<sup>[6,7]</sup>, the experimental change in potential with molar composition steepens between the 1.6:1 and 2.2:1 molar ratios and more closely follows the form of equation (8). Obviously, this model is inappropriate for aluminum chloride:1-butylpyridinium chloride ratios in excess of 2:1. In addition, the estimation of the equilibrium constant in this manner assumes that the positive shift due to the mixed potential arising from the spontaneous reduction of the 1-butylpyridinium cation by elemental aluminum is not significant (<1.0:1.0 mole ratio range). This will be true if the exchange reaction for the aluminum couple is large relative to the cathodic reduction currents; however, the magnitude of  $K'_3$  should be regarded as a minimum value. Figure 2 illustrates the changes in the species composition calculated for the  $K'_3$  value of  $3.83 \times 10^{-13}$ . The chloride ion mole fraction almost spans the range found in the proton/hydroxyl equilibrium in water.

Potentiometric curves were obtained at 30°C, 60°C, 120°C and 175°C but did not reveal any significant differences in the potential span. This is unlike the  $\text{AlCl}_3\text{:NaCl}$  system at higher temperatures which has a 25% reduction in the potential span at 250°C compared to that at 175°C.<sup>[7]</sup>

### DISCUSSION

The results of this potentiometric study are consistent with the Raman spectral data and indicate that the formation of  $\text{Al}_2\text{Cl}_7^-$  ion proceeds to a greater extent in the  $\text{AlCl}_3$ :1-butylpyridinium chloride system than in the previously studied alkali metal halide systems (Table I). Unfortunately, it is difficult to compare directly the absolute values for the equilibrium constant  $K_3$ . Different model systems have been used and in certain cases there are restrictions in the accessible stability of liquidus temperature ranges. Torsi and Mamantov<sup>[11]</sup> have reported an increase in  $K_3$  through the cation series  $\text{Li}^+ < \text{Na}^+ < \text{K}^+ < \text{Cs}^+$ , at 400°C. This finding essentially agrees with the interpretations by Ikeuchi and Krohn<sup>[12]</sup>, or Schulze *et al.*<sup>[8]</sup> who use a 3-parameter model and report a decrease in the equilibrium constants for reaction (4) at 450°C;  $\text{Na}^+(2 \times 10^{-6}) < \text{Rb}^+(1.1 \times 10^{-8}) < \text{Cs}^+(1 \times 10^{-9})$ ;  $K_3$  for this model remains relatively constant (Table I). The direct correlation between the cationic radii and extent of  $\text{Al}_2\text{Cl}_7^-$  ion formation may relate to the inverse ionic field strength as one might expect anionic complexes to be more stable with decreasing cationic polarizability<sup>[11]</sup>. The value of  $K_3$  obtained with the relatively large, organic 1-butylpyridinium cation is consistent with this general trend.

The major effect of temperature variation, from 30°C to 175°C, is consistent with a change in the reaction equilibrium, according to the relation

$$\Delta G = -RT \ln K_3.$$

On the basis of the equilibrium constants obtained from the potentiometric curves for this system (Table I), the free energy remains approximately constant throughout the 145°C temperature span at  $(7.1 \pm 0.3)10^4 \text{ J mol}^{-1}$ . Unfortunately,



precise thermodynamical data is precluded because of the corrosion process which occurs in the basic  $\text{AlCl}_3$ :1-butylpyridinium chloride systems. One practical consequence of the increased  $\text{Al}_2\text{Cl}_7^-$  ion formation and the lower activity of free  $\text{Al}_2\text{Cl}_6$  in these molten mixtures, relative to the  $\text{AlCl}_3$ :NaCl system, is that sublimation losses of  $\text{Al}_2\text{Cl}_6$  are minimal even at  $175^\circ\text{C}$ . They may be also useful solvent systems for stabilizing unusually low valence metallic ion species.

#### Acknowledgements

This work was supported by the Office of Naval Research under contract N00014-77-C-0004.



TABLE I

Mole fraction equilibrium constants for  $\text{Al}_2\text{Cl}_7^-$  ion formation in  $\text{AlCl}_3$ : M Cl<sup>a</sup> melts from potentiometric data.

System	$K_3^b$	Temperature/°C	Reference
$\text{AlCl}_3$ :1-butylpyr. Cl	$<3.83 \times 10^{-13}$	30	This work
"	$<5.66 \times 10^{-12}$	60	"
"	$<3.61 \times 10^{-10}$	120	"
"	$<1.19 \times 10^{-8}$	175	"
$\text{AlCl}_3$ :NaCl	$1.06 \times 10^{-7}$	175	[6]
"	$8.00 \times 10^{-8}$	175	[7]
"	$7.77 \times 10^{-8}$	175	[10]
"	$1.33 \times 10^{-7}$	175	[9]
$\text{AlCl}_3$ :LiCl	$1.6 \times 10^{-4}$	400	[11]
$\text{AlCl}_3$ :NaCl	$1.0 \times 10^{-5}$	400	[11]
$\text{AlCl}_3$ :KCl	$1.6 \times 10^{-6}$	400	[11]
$\text{AlCl}_3$ :CsCl	$4.0 \times 10^{-8}$	400	[11]
$\text{AlCl}_3$ :NaCl	$5.78 \times 10^{-7}$	450	[8]
$\text{AlCl}_3$ :RbCl	$2.87 \times 10^{-7}$	450	[8]
$\text{AlCl}_3$ :CsCl	$7.58 \times 10^{-7}$	450	[8]

a. M represents a cationic species.

b. Dissociation constant for  $2 \text{AlCl}_4^- \rightleftharpoons \text{Al}_2\text{Cl}_7^- + \text{Cl}^-$

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Figure 1

Potentiometric data for  $\text{AlCl}_3$ :1-butylpyridinium chloride versus  $\text{Al}(0)/2:1$  mole ratio mixture reference; x  $30^\circ\text{C}$ , o  $60^\circ\text{C}$ , ●  $120^\circ\text{C}$ ; — theory ( $30^\circ\text{C}$ ).



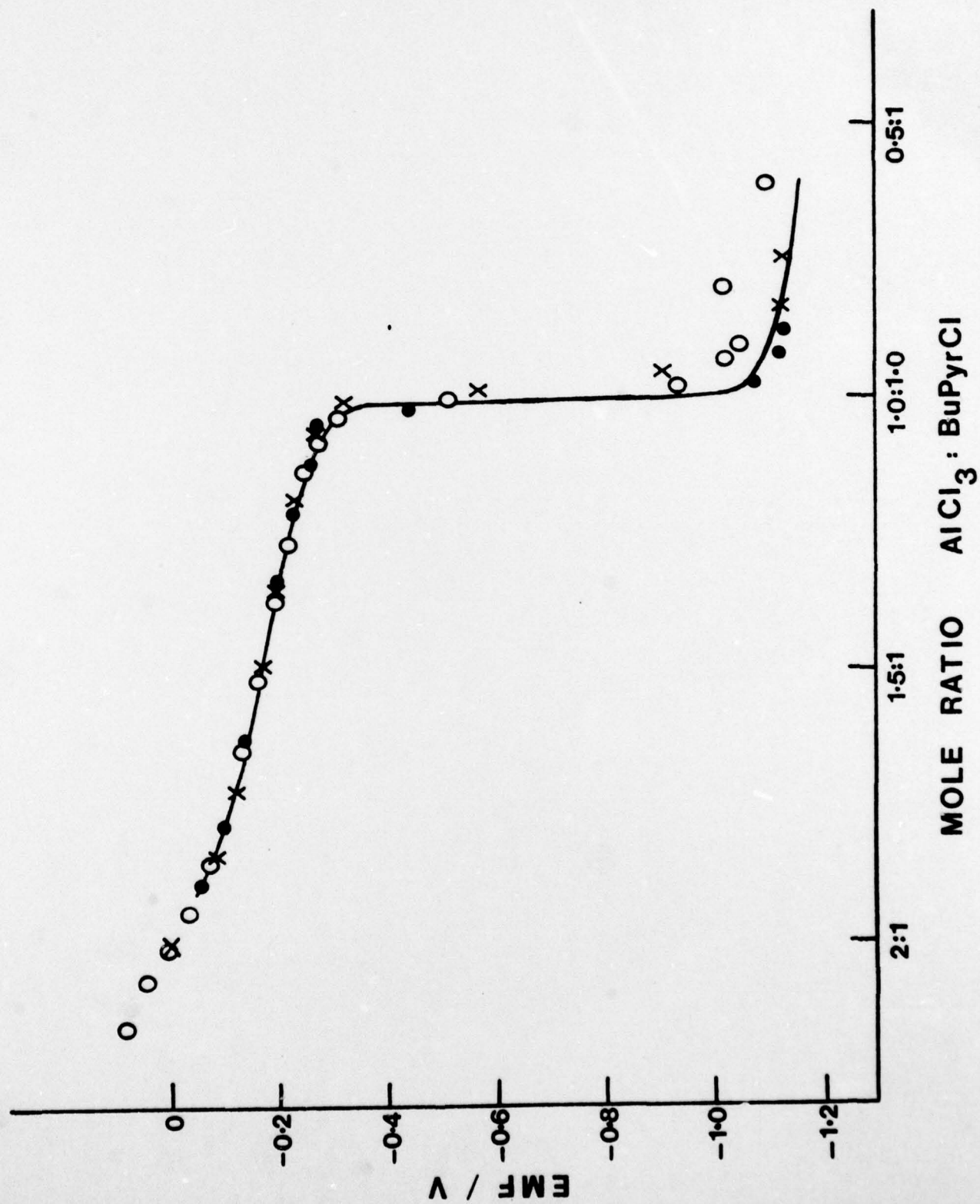
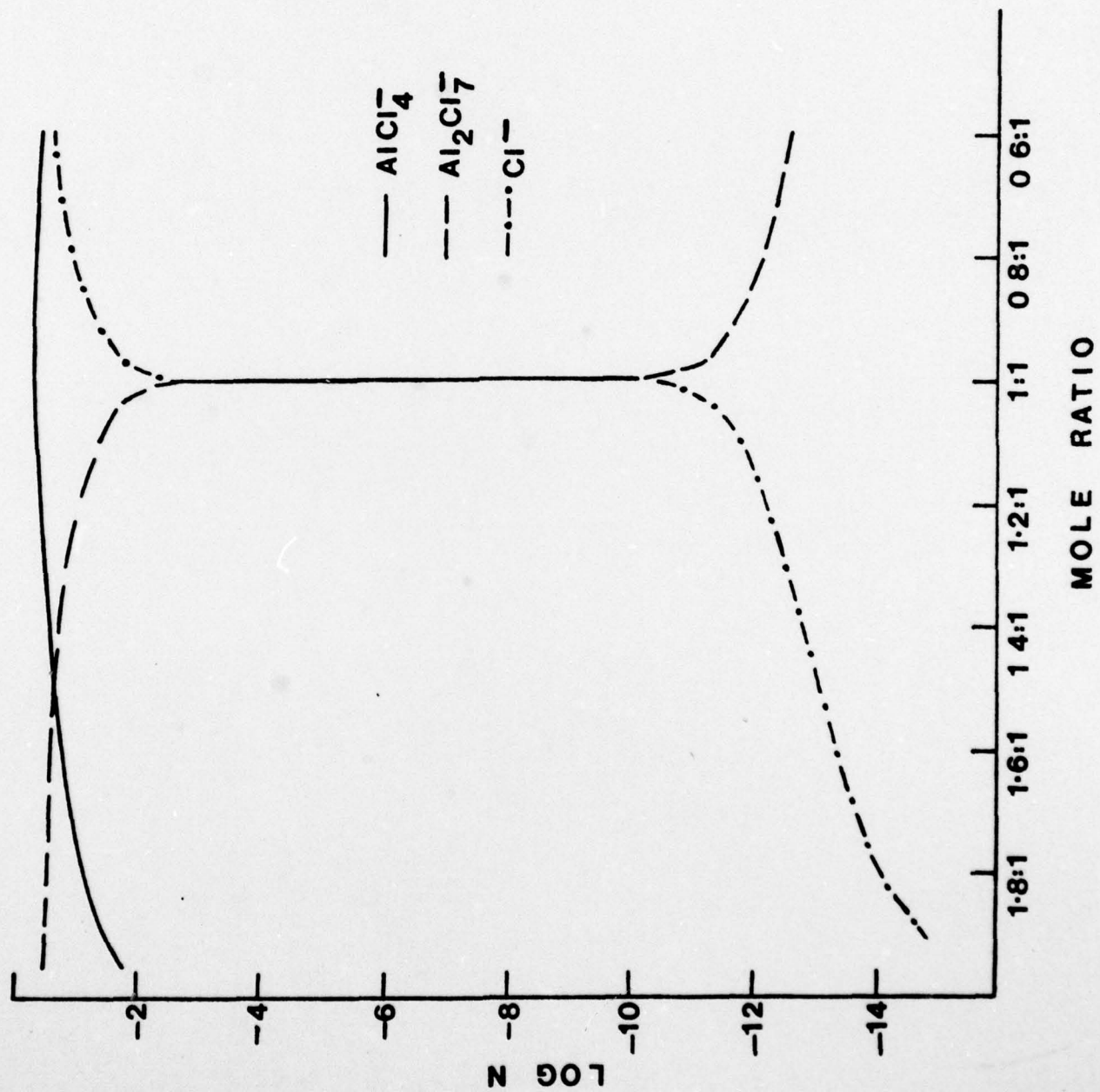




Figure 2

Mole fraction,  $N$ , of the major species in the melt at 30°C as a function of the net  $\text{AlCl}_3:\text{RCl}$  mole ratio.



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